SUMMARY OF THE KILOROD PROJECT — A

SEMIREMOTE 10-KG/DAY DEMONSTRATION OF

$^{233}UO_2$-ThO_2 FUEL-ELEMENT FABRICATION BY THE

ORNL SOL-GEL VIBRATORY-COMPACTION METHOD

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1. ABSTRACT

Heretofore, the development of a reactor fuel cycle based on thorium and ^{233}U had been hindered by radioactivity resulting from the ^{232}U decay daughter products normally present in ^{233}U. Because of this associated activity, an economical thorium-^{233}U fuel cycle requires rapid, simple chemical and mechanical processes easily adaptable by nature to remote operation. The ORNL-developed sol-gel process for preparing granular, mixed oxides of thorium and uranium uniquely meets this requirement. A complete system for making fuel elements is obtained by coupling the sol-gel process with vibratory-compaction loading of fuel tubes. During the past year an intermediate-scale demonstration (10 kg a day) showed the feasibility of this combination of sol-gel and vibratory compaction. During this demonstration, more than 1 metric ton of 3% $^{233}UO_2$ — 97% ThO_2 solids were prepared. Approximately 1000 fuel rods (hence the name Kilorod Program) were fabricated from the solids to fill a request of the Brookhaven National Laboratory.

The Kilorod Operation was made up of three straightforward steps: (1) preparation of feed materials, (2) the sol-gel process, and (3) fuel-rod fabrication. In the preparation of feed materials, the ^{232}U daughter products were removed from the ^{233}U by a single-cycle solvent extraction process, and thorium nitrate crystals were converted to a dispersible ThO_2 under a superheated steam atmosphere at 450 to 500°C. In the sol-gel process, $UO_2(NO_3)_2$ and ThO_2 feed stocks are blended at 80°C to form a stable sol (3% $^{233}UO_2$ — 97% ThO_2). The sol is dried to a gel at 80°C and then calcined and reduced at 1150°C to produce a sintered, fragmented UO_2-ThO_2 product. Fuel rods are then fabricated by crushing the sintered solids and screening the solids through a 6-mesh onto a 16-mesh screen. The remainder of the solids are ball-milled to a powder of "smeared" size distribution. These powders are then blended in proper proportions, loaded into Zircaloy tubes by vibratory compaction, and the rod end-fixture is welded into place. The completed fuel rod is then inspected to ensure concentricity, straightness, weld integrity; finally it is decontaminated.

All mechanical and chemical processes of the Kilorod Program behaved as expected. After the startup period,

equipment-repair frequency was low. Both feed-preparation operations realized 100% on-stream efficiency. On-stream efficiency was 90% for the sol-gel operation for the entire operating period and 80% for the rod-fabrication equipment after startup. The latter two are particularly good for directly maintained high-level radiochemical operations.

Fifty kilograms of ^{233}U (containing 38 ppm ^{232}U) were purified by a new solvent extraction system (2-1/2% di-$\underline{sec}$-butyl phenyl phosphonate in diethylbenzene). The extracted product met desired chemical specifications for removal of ^{232}U daughter products.

More than 1200 kg of ThO$_2$ suitable for use in the sol-gel part of the process were prepared by hydrothermal denitration. Over 1 metric ton of ^{233}UO$_2$-ThO$_2$ meeting established chemical specifications was prepared by the sol-gel process during the program.

Nine hundred rods, each containing 890 g of mixed oxide, and 200 rods, each containing 310 g of the same mixed oxide were prepared. The fuel column in these rods averaged 89.6% of theoretical density, and variations of density were held to within ±2% of a median value along the active length of the fuel column. The entire process may readily be scaled to larger operations.

Experience was obtained in the handling of large quantities of ^{233}U, and radiation data necessary for the design of larger facilities and higher activity levels were collected. Radiation exposure to workers in routine operations was limited to a tenth of the permissible levels. Indeed, a single batch, approximately 100 times more radioactive than the routine Kilorod batch, was prepared to measure exposures to workers at extreme levels of activity. In this safety test, aged ^{233}U was used to simulate 14-day-old feed solution containing 800 ppm of ^{232}U. The resulting exposures, using normal Kilorod operating procedures, were at worst eight times higher than when the customary Kilorod ^{233}U feed solutions had been used. Thus, the present equipment, procedures, and working areas can provide radiological safety at much higher concentrations of ^{232}U than that used in the present program.

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2. INTRODUCTION

This summary report describes a successful engineering demonstration of the sol-gel, vibratory-compaction process developed here, and its application to the production of 1100 fuel rods loaded with ceramic-grade thoria-urania for use at Brookhaven National Laboratory. This demonstration, conducted in the Kilorod Facility, represents the first

remote fabrication of ceramic-oxide fuels. The facility was shielded to protect the operators from the gamma radiation associated with ^{232}U and ^{228}Th, the contaminants in ^{233}U fuels.

The sol-gel portion of the process, while developed primarily for recycling thorium reactor fuels, promises to become an almost universal method for preparing ceramic fuels because of its simplicity and flexibility. In recycling ^{233}U and thorium fuels by the sol-gel process, workers can be more easily protected from the gamma rays emitted by the decay products of ^{232}U and ^{228}Th. Conventional methods, besides being more complex and less versatile, require much higher calcination temperatures to densify the product and are not so adaptable to remote operation in a shielded facility.

The Babcock and Wilcox Company has installed the sol-gel portion of the process in their pilot plant at Lynchburg, Virginia, for producing ^{233}U-Th recycle fuels. They will use this process along with vibratory packing for recycling such fuels to the Spectral-Shift-Control Reactor, for example.

Since about a thousand fuel rods were to be made, the program was called the "Kilorod Program." The facility contained equipment to:
1. purify ^{233}U by solvent extraction,
2. prepare a mixed thorium-uranium dioxide by the sol-gel process,
3. size the powdered oxide,
4. load it into tubes by vibratory compaction,
5. weld the end plugs, and inspect the finished rods.

This summary report on the engineering-scale demonstration of the sol-gel and rod-fabrication processes includes the following: description of the pilot plant and procedures, operating experience, radiation experience, conclusions and recommendations, plus an Appendix, where certain detailed information can be found.

3. DESCRIPTION OF THE KILOROD PILOT PLANT

The flowsheet for the Kilorod complex is shown in Fig. 1. Except for the denitration operation, which is located in a separate building

3.1.1.2 The Kilorod Design and Radiation-Control Policy. — A single
cell (No. 4) of Building 3019 was renovated to receive the pilot plant
equipment. Two balconies were erected, and processing cubicles were built
on each balcony and the main floor (Fig. 3). All the high-level alpha-
gamma work was done in these cubicles. Alpha containment is effected by
lining the inside of all processing cubicles with 1/8-in. mild-steel sheet.
Shielding from the gamma rays is provided by either 4-1/2-in. steel plate,
or 8-in. barytes concrete block. Gloved hands or Castle-type manipulators
are used for all operations. High-density glass windows were provided for
observing operations at all work (glove) stations. Conventional "bag-out"
stations were installed for introducing or removing materials from the
cubicles. Retractable lead shields were provided for closing glove-port
openings when gloves were not in use. Fire shields were also provided to
protect the gloves during periods when work was not underway. These shields
were set in place on the inside surface of the alpha membrane as the glove
was removed from the hand.

In the design and location of the equipment for this high-level alpha-
gamma operation, attention was given to the repair of equipment considered
susceptible to mechanical failure. Accordingly, equipment considered sus-
ceptible is located near a glove and/or bag-out station, or such a station
is provided. In this way, replacement parts could be "bagged" into the
contaminated area, and replacement or repair can be carried out without
making entry into the cubicle. The design time required for this was well
spent since minor maintenance (replacement of pH electrodes, thermocouples,
lights, or adjustment of limit switches) could be done promptly and as
needed without requiring a plant shutdown.

3.1.2 Preparation of Feed Materials Entering the Sol-Gel Process

In essence, preparing the feed material consists of two processes:
ThO_2 powder is prepared by the hydrothermal denitration of thorium nitrate
crystals, and ^{233}U as a nitrate solution is freed of ^{232}U daughter pro-
ducts by solvent extraction.

3.1.2.1 Thorium Oxide Feed Preparation: An Integral Part of the
Sol-Gel Process and the First Step Toward Preparing the Mixed Oxides
(ThO_2-UO_2) Needed for Vibratory-Compaction Feed. — The thorium oxide feed
is prepared by hydrothermal denitration of thorium nitrate tetrahydrate
(TNT) in a horizontal rotary denitrator. The calciner shell in which the
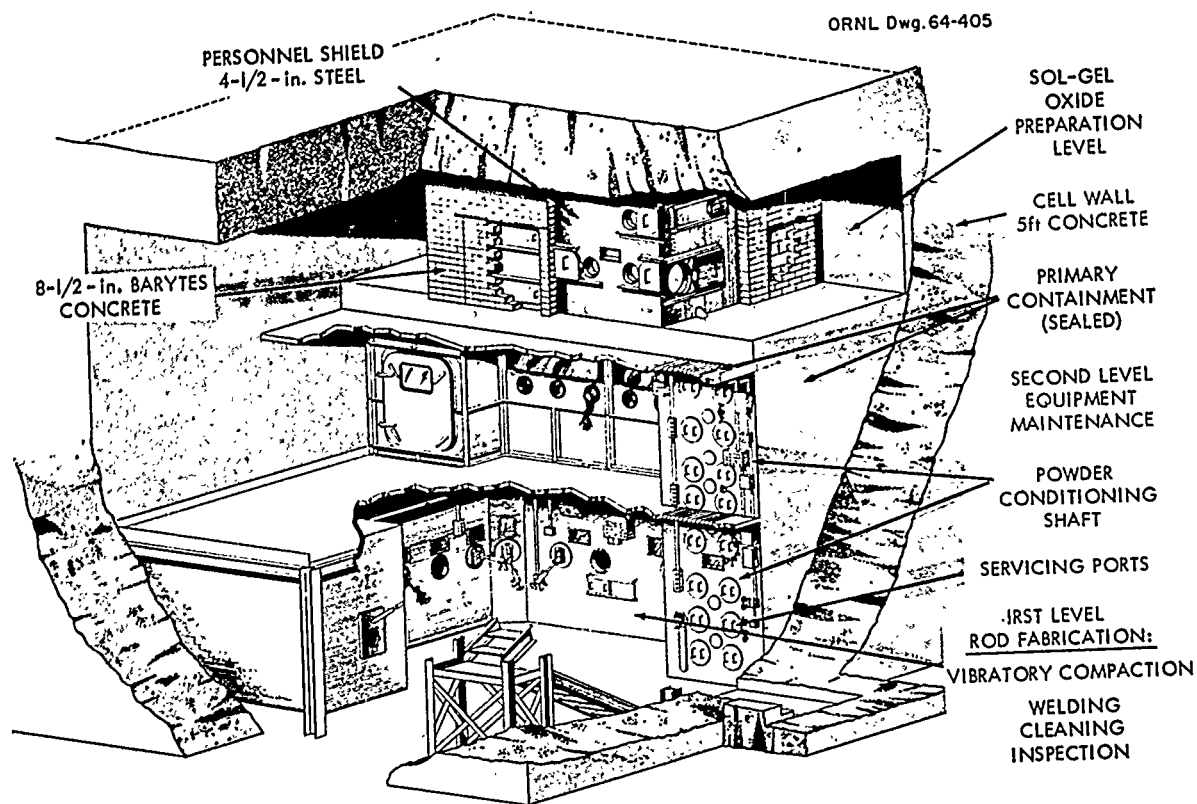
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Fig. 3. Kilorod Solids-Preparation and Rod-Fabrication Facility.
The process flow begins at the top left with sol blending, flows to the
right where the solids are fired, passes down the vertical shaft as the
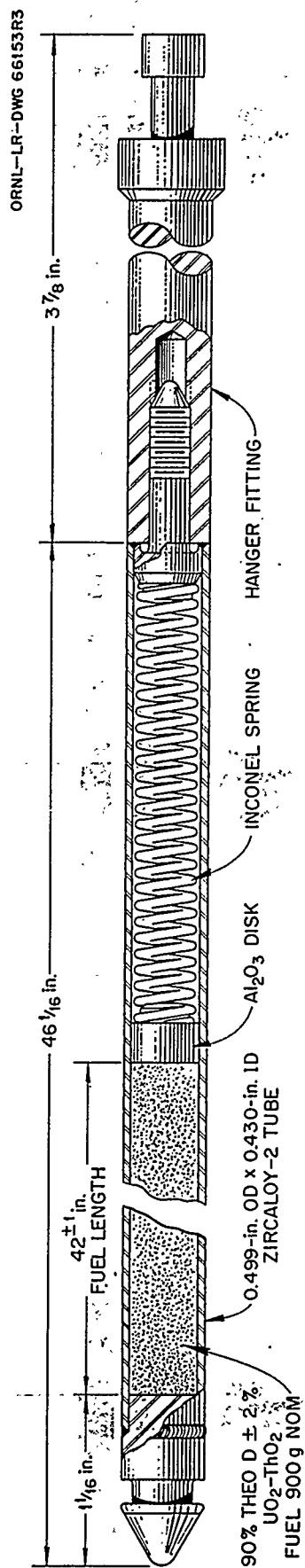powder is prepared, and across the bottom as the rods are loaded and tested.

Fig. 23. Design Features of the Brookhaven National Laboratory Fuel Rod.

Sump Pumps. — Since the first-level cubicles were located below the hot-drain system, the liquid waste was raised from the sumps to the hot drain by steam-jet pumps. The hot-drain system was thus left directly connected to the cubicle atmosphere through the jets. Because of this fault, it was not possible to attain the required cubicle in-leakage rate. Each delivery line was opened and a valve inserted to correct this condition.

At the end of the cold startup operations all equipment that would contain ^{233}U was cleaned carefully to remove all traces of the depleted uranium. All portable equipment inside the cubicles and all saggers in which depleted uranium had been fired were discarded. The cubicle and the floors and walls were scrubbed thoroughly. All samples of uranium-bearing solids were removed to another building (the denitrator area), sealed in a large carton and stored.

Nine of the first ten batches prepared were sampled before loading into the fuel rods and submitted for mass assay to ensure that no isotopic dilution had occurred. No perceptible dilution was found.

3.3 Utilities and Waste Disposal

3.3.1 Emergency Power and Lighting

Radiation instruments and lights provided within the Kilorod cell are connected to both normal and emergency power supplies. A constantly charged, battery-powered light was also provided in the event that the emergency generator did not start. No other equipment was connected to the emergency power since operator safety was not involved.

3.3.2 Air Flow and Cubicle-Cell Operating Pressures

Air (2400 cfm) entered the cell by way of the normal access stairwell at "Point A" on Fig. 27. Before reaching point A, the air passes through a filter bank, equipped with gravity-operated louvres that act as back-flow preventers. The filter was common, pleated, glass wool. This filter bank-louvre system and the cell proper constitute the outer, or secondary containment for the operation. The operating cubicles and absolute air filters (see below) on the cubicles formed the primary (high-level alpha) containment.
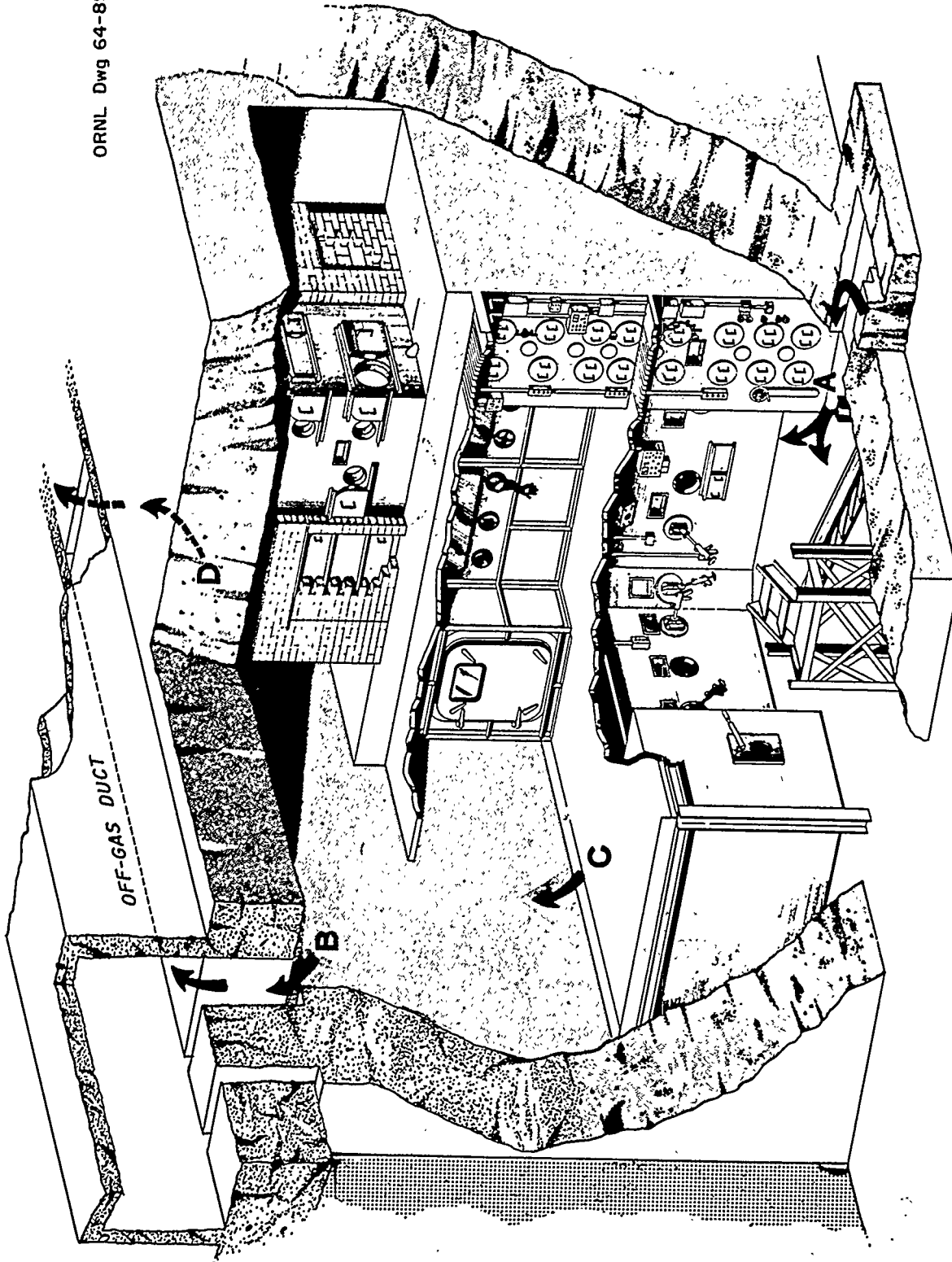
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Fig. 27. Air-Flow Pattern in Cell 4.

Within the cell, the air is split into two streams: One supplies air to the working, or occupied, portion of the cell exterior to the process cubicles, while the other supplies air to the processing cubicles. The first of these air streams passes up through the open portion of the cell shaft and exhausts through an absolute filter bank located in the top northwest corner of the cell at "Point B." This filter bank is also equipped with gravity-operated louvres. The discharge leads into the building off-gas duct and thence to the stack (Building 3020). The second air stream enters a bank of absolute filters in the service area of the first balcony ("Point C"). From this bank, ducts equipped with gravity-operated louvres conduct the air into the process cubicles. Leaving the process cubicles, the air passes through lever-operated damper valves at the air exit port of each cubicle and then through a final bank of filters. This bank includes both roughing (Fiberglas, 1 in.) as well as absolute filters. Again, these filters are located on the first balcony service area. From the filter bank, the air passes through the upper, northeast corner of the cell ("Point D"), through gravity-operated louvres into the building off-gas duct and stack. The air passing through the working space was regulated at about 2200 cfm, while the total quantity passed through all process cubicles at about 200 cfm.

The pressure in the occupied areas of the cell was always held at 0.1 in. (H_2O) below atmospheric pressure. The pressure within the operating cubicles was held to 0.1 to 0.2 in. H_2O below the pressure within the cell; thus the operating cubicles were at a pressure 0.2 to 0.3 in. (H_2O) below atmospheric. The building off-gas duct provided the pressure drop needed to maintain the negative pressure differences. The flowing air proceeded from relatively uncontaminated to progressively more contaminated ones. For example, on the first floor, air moved from the carrier-loading station through the various cubicles of the fabricating area and was removed at the rod-loading station.

Immediately before the start of cold-testing, each cubicle was made ready, and the air in-leakage rate was determined. All hot working spaces met the <0.005 working space vols/min in-leakage rate (at 1 in. H_2O negative pressure), required by Laboratory practice.[9] The filter banks were tested with dioctyl phosphate smoke in situ, also as required by Laboratory practice, and found to be >99.99% efficient.

Fig. 27. Air-Flow Pattern in Cell 4.

3.3.3 Disposal of Wastes

Each of the processing cubicles was equipped with a floor drain, or if on the bottom floor, a steam jet to the hot waste system. All these waste lines and the process condensate from the sol-gel operations was piped to the hot waste. Normal steam condensate and cooling water were "tied" to the process waste header.

Each vessel in the sol-gel system was connected to the hot-vessel off-gas system and equipped as well with a water-cooled condenser. An inward sweep of air was maintained on all vessels at all times. Gases pulled through these condensers were piped to the common hot-off gas header, passed through an absolute filter to remove particles, and then into the hot-off-gas disposal system for the plant.

Solid wastes were picked up by vacuum cleaner from the tables and other working surfaces. As the bags were filled, they were replaced, and the full ones bagged out for reprocessing of the material they contained. Other solid wastes (broken crucibles, etc.) were bagged out and buried.

4. OPERATING EXPERIENCE

4.1 Construction and Cold Startup Operations

The SX equipment was converted by September 1962, and cold testing started in November. The first hot (trace-element) runs were made in January 1963 and the plant placed in operation for the Kilorod Program in March.

Installation of the equipment began in cell 4 in mid-October 1962, and was completed in early March, 1963. Preliminary equipment testing began on February 15 and was completed on May 3. During the period of April 3 to May 3, test results showed that several equipment changes would have to be made in order to meet the throughput rates for which the sol-gel process was designed (see Sec 3.2.2). These changes were completed and final adjustments made by May 10. Necessary equipment changes and cold-testing operations were completed on the rod-fabrication equipment in early June. Hot feed was introduced into the sol-gel cubicle on June 11, 1963.

4.1.1 Preliminary Testing of the Solvent Extraction Equipment and Flowsheet

Upon the completion of the necessary changes to the Thorex Pilot Plant, the solvent extraction plant was operated in four trace-level and two

Table 26. Kilorod Program Solvent Extraction
Over-All ^{233}U Material Balance

Input	^{233}U (g)
From U-60	7,081
From storage bottles	599
From Davison Chemical Company	25,179
From Jezebel fuel	12,026
From M. Lloyd	76
From bottle 36P	64
Total input	45,035

Output	
To sol-gel	26,779
To Bettis	12,989
To M. Lloyd	107
To Y-12	142
To BTC storage	127
Accountable losses	97
Retained in product tank	3,932
Retained in feed tank	595
Total output	44,768

Unaccountable Losses, g   267

Material Balance

$$\frac{44,768}{45,035} \times 100 = 99.4\%$$

Table 27 is a summary of the ^{233}U and thorium material balances obtained in the individual solvent extraction runs. The ^{233}U material balances varied from 96.81 to 102.66% and averaged 98.98%. The wide variation and relatively inferior balances obtained in several runs is thought to be due to system holdup, an estimate of which is impractical. The satisfactory over-all balances obtained for the entire program is thought to be a more accurate indication of the successful measurement of input and output quantities.

6. CONCLUSIONS AND RECOMMENDATIONS

The Kilorod Program was an unqualified success, meeting every objective. The 10-kg-per day design rate for the sol-gel operation was sustained, as scheduled, over long operating periods. The longest of these was fourteen consecutive days in the November-December period. All equipment except the calciner could produce at least double the production design rate. Rod-fabrication operations were similarly successful, with an average sustained production rate of 13 rods having been demonstrated over a period of 10 scheduled operating days. A maximum of 21 rods was fabricated in a single working day, once during the program. All chemical and fabrication specifications were met or exceeded.

From the Kilorod experience, the following conclusions are reached:

1. The sol-gel process for preparing a 3% UO_2 — 97% product is now a practical, working process. This process can be scaled up directly to any immediately foreseeable production rate.

2. A sol-gel, rod-fabrication combination is also a practical, working process that can be scaled up directly to any immediately foreseeable production rate.

3. Specifications requiring both high product purity and fuel element uniformity can be met routinely in a combined sol-gel, vibratory-compaction, fuel-rod-preparation plant.

4. Batch rejection and internal recycle of materials (as wastes) are almost nil in a sol-gel, vibratory-compaction operation as represented by the Kilorod installation.

5. Both sol-gel and rod-fabrication operations are amenable to rigid analytical control, as evidenced by the process control during this program.

6. Exposure of workers to radiation at the 38-ppm ^{232}U level was far below permissible limits.

7. Considerably higher ^{232}U concentrations can be handled safely in the present equipment by current operating procedures.

8. Loss of process materials in all operations can be held to extremely low if not insignificant values.

9. Aged ^{233}U (38 ppm ^{232}U) can be satisfactorily purified for use in the sol-gel process by a solvent extraction process based on extraction by di-sec-butyl phenyl phosphonate.

From Kilorod experience the following recommendations are made:

1. The problems associated with the gases contained in the final sol-gel product should be studied. Unfortunately, it was impossible to study them in conjunction with the normal operation.

2. Consideration should be given to the design, construction and startup of a continuous denitrator. The present unit, even though completely dependable in all respects, is a high-labor-cost operation. Any future, large-scale thorium processing plant must necessarily include a complete and continuous conversion of a thorium nitrate solution to a powder (or sol). In this innovation lies probably the largest single cost-reduction item now apparent and is also probably the one requiring the least effort (or money).

3. The cost of gases (Ar, Ar--4% H$_2$) was excessive and must be reduced. Other gases, quantity purchase and storage, and error-proof mixing system should be considered.

The success of the Kilorod Program represents a significant breakthrough in the fabrication of ceramic fuel elements. The sol-gel, vibratory-compaction combination offers advantages in simplicity, reliability, and ease of process control available in no other fabrication scheme.

7. ACKNOWLEDGMENTS

The authors wish to acknowledge the contribution made by many people to the success of the Kilorod Program and the preparation of this report. Particular appreciation is expressed for the guidance of F. L. Culler, D. A. Douglas, Jr., J. C. Bresee, D. E. Ferguson, R. E. Brooksbank, J. M. Chandler, A. L. Lotts, and J. D. Sease.

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Appreciation is also expressed to R. H. Rainey, A. D. Ryon, and M. G. Baillie (loanee from Australia) for their contribution to the success

8. APPENDIX

8.1 Detailed Information on the Solvent Extraction Operation

8.1.1 Description of Facility

The Thorex Pilot Plant in cells 5, 6, and 7 of Building 3019 was modified to provide a facility for purification of the ^{233}U feed stocks for the program. A schematic equipment flowsheet is presented in Fig. 1A. The ^{232}U content and age of the ^{233}U available, together with the planned semiremote method of fabrication, indicate that a gross gamma decontamination factor of only 100 was required. In addition, the sol-gel process requires that the ^{233}U feed should not have a nitrate to uranium ratio above 2.5 if a satisfactory gel is to be obtained. This requirement necessitates a thorium decontamination factor of at least 1000.

Extensive experimental work by Ryon indicates that the use of a solvent containing 2.5% (di-sec-butyl phenyl phosphonate), also in diethyl-benzene, provides gross gamma and thorium decontamination factors as much as 4 times greater than that offered by a similar system employing tributyl phosphate. While it is true that the decontamination-factor requirement is relatively low, it was decided to use the newer DSBPP solvent to obtain pilot plant experience.

In the DSBPP flowsheet, the adjusted feed is introduced into the extraction column where it is contacted with the extractant. The uranium is extracted into the organic phase, which is then scrubbed with a 0.8 $\underline{M}$ aluminum nitrate solution which is 0.4 $\underline{M}$ acid deficient to remove entrained thorium and ionic contamination. The aqueous raffinate from the extraction column is routed to storage. The scrubbed organic then cascades to a strip column where the uranium is removed from the organic phase with water. The resultant uranium solution is then transferred through a static diluent wash column for the removal of trace quantities of organic prior to concentration. Spent solvent is collected batchwise, treated with 0.2 $\underline{M}$ Na$_2$CO$_3$ and reused.

To provide a facility for the ^{233}U purification the Building 3019 solvent extraction facility was extensively reconditioned and modified. Extracting and scrubbing were done in the original 5-in.-diam, 38-ft-high
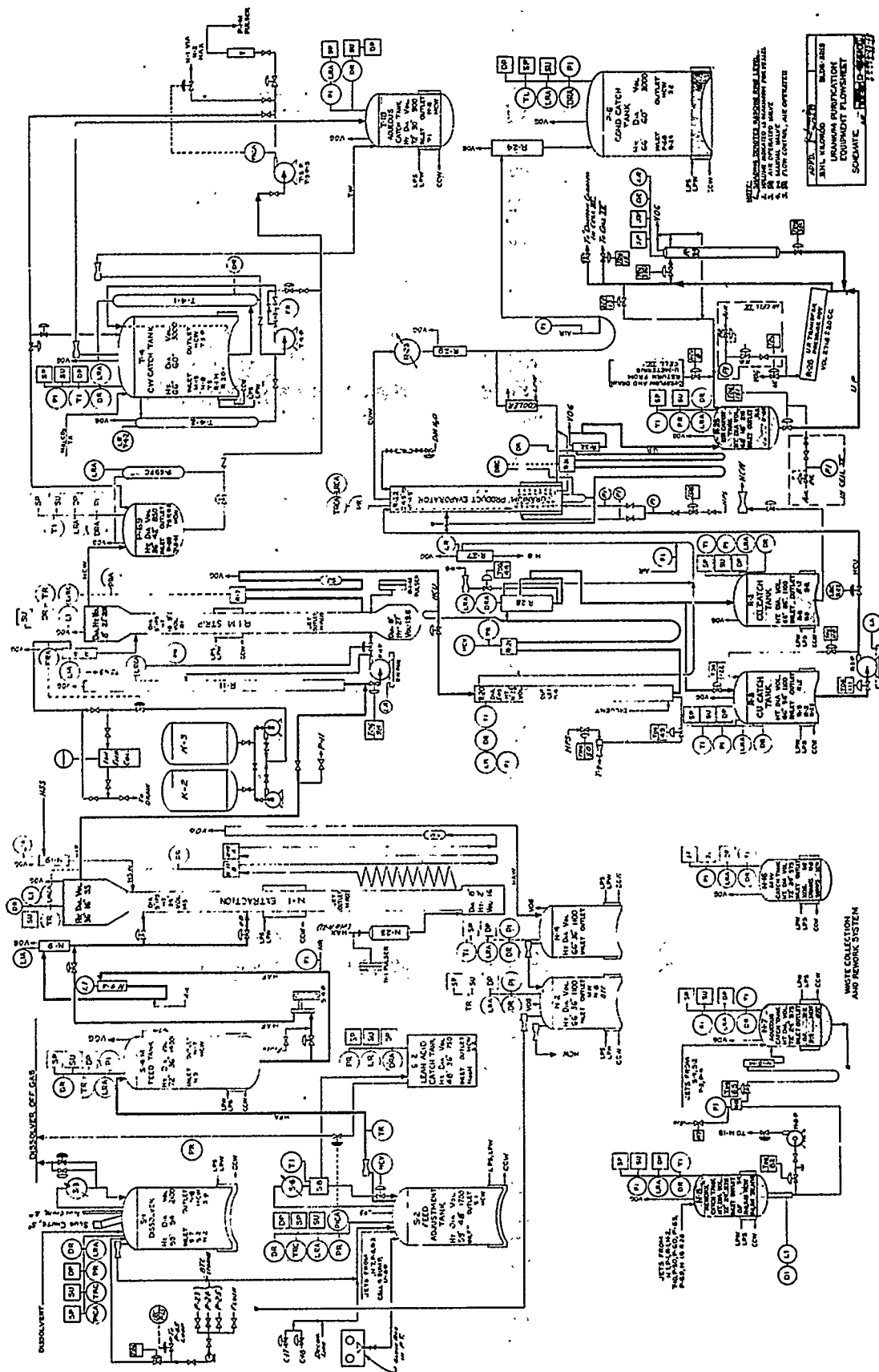
Fig. 1A. Uranium Purification Equipment Flowsheet.

pulsed column (N-1). Laboratory results indicate that at least four extraction and three scrub stages will be required. When operating at a pulse amplitude of 1 in. and a frequency of 58 cpm, the required stages are obtained when introducing the feed at a point to obtain 15 ft of extraction and 17 ft of scrub. If necessary, additional scrub stages could be obtained by operating with an additional 5-in.-diam column providing 12 ft of pulse plates (P-1).

The organic stripping was performed in an existing 5-in.-diam, 20-ft-high column (R-1). Ryon's results indicate that when operated at 1.0-in. amplitude and 50 cpm the required two stages will be obtained.

A new 2-in.-diam column packed with 1/2-in. Raschig rings (stainless steel) for the static diluent-wash column, and a new 5-in.-diam product evaporator and product storage tank, were installed.

The majority of the vessels to be used are not of critically safe geometry; consequently, several important operational and equipment modifications have been made. The total mass of uranium within the facility did not exceed 8 kg. Solutions were maintained below the ^{233}U maximum "eversafe" value of 10 g/liter by flowrate adjustments for aqueous streams and chemical saturation for organic streams. The only stream which exceeded this value was the final product stream (100 g/liter), which was boiled down in a 5-in.-diam evaporator critically safe to a concentration of 200 g/liter.

In addition to mass and concentration control, the feed extraction column and raffinate catch tank contained sufficient thorium to yield a ^{233}U/Th ratio of <0.025.

As a final safety device, Pyrex Raschig rings containing 4% B were placed in critical process vessels, notably in the expanded sections of the columns, waste tanks, solvent tanks, and product tanks.

The product evaporator is made of 5-in. pipe, with a jacketed section providing 4.5 ft^2 of heating surface. Three perforated plates are provided for vapor de-entrainment, and the feed is introduced between the bottom two plates. Cold tests before installation indicated that at 25 liter/hr boilup, entrainment was negligible when operated with no reflux; however, in the intermediate-level runs, operation without reflux resulted in overhead losses as high as 0.5%. The use of 3 liter/hr water reflux introduced 2 ft above the feed reduced the loss to <0.01% during the latter part of the intermediate level run and was negligible throughout the hot operation.

As previously mentioned, satisfactory sol-gel product is dependent on strict control of the nitrate ion concentration in the sol. This establishes an upper limit on the nitrate-to-uranium ratio in the solvent extraction product. (In the Kilorod process this ratio is 2.5.)

Product from the solvent extraction system normally contains a constant concentration of excess nitrate ion (nitrate from sources other than uranyl nitrate). As a result, the nitrate-to-uranium ratio is high at the beginning and end of a solvent extraction run since the uranium concentration is low at these times. Consequently, off-specification product is obtained for a period at the beginning and at the end of each run.

During operations, a product cut is collected as feed to the evaporator. The nitrate-to-uranium ratio is controlled by diversion of product between the product tank and recycle tank.

8.1.2 Detailed Discussion of Operating Procedures

Operation of the Kilorod sol-gel and rod-fabrication facility at the design capacity required about 265 g of ^{233}U per day and a program total of nearly 28 kg. The capacity of the solvent extraction facility at equilibrium flowsheet conditions is 250 g/hr. Although the capacity of the solvent extraction facility is such that it would be possible to purify the entire ^{233}U requirement in about 112 hr of equilibrium operation, ^{232}U daughter growth necessitates intermittent operation. Initial prediction of this growth and criticality safety considerations led to the decision to accumulate no more than a three weeks' supply or to process the uranium in batches of about 5 kg.

Each purification batch involved the following operations:

1. cold feed and scrub makeup,
2. metal dissolution and/or hot feed makeup,
3. solvent extraction,
4. solvent treatment, and
5. reworking of "off-spec" solutions.

These operations were conducted with two operators per 8-hr shift, following detailed operating sheets. Operations involving significant quantities (more than 500 g) of ^{233}U were supervised by technical personnel. The run sheet for the solvent extraction operation is presented as an example of the procedures used.

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